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(54) CAST STRIP FOR R-FE-B MAGNETIC ALLOY AND ITS PRODUCTION

(57)Abstract:

PURPOSE: To prevent deterioration of the degree of orientation by regulating the thickness of an alloy, having a composition composed essentially of specific amounts of R, B, and Fe and also having a homogeneous structure composed of R2Fe14B type crystals of specific grain size and R-enriched phases, to a specific value.
CONSTITUTION: A molten alloy, composed essentially of, by atom, 10-25% R, 2-15% B, and 60-88% Fe, is cooled by means of a rapid cooling roll from a temp. between (liquidus temp. +5) and 300°C down to 700-1000°C cast strip temp. at (2×103 to 7×103)°C/sec primary cooling rate. After detached from the roll, the cast strip is cooled down to a temp. not higher than the solidus temp. at (50 to 2×103)°C/min secondary cooling rate. A fine structure, in which R2Fe14B type dentrites or columnar crystals of 3-15μm average minor axis crystalline grain size, containing fine crystals of <1μm average minor axis crystalline grain size by ≤10%, and R-enriched phases of ≤5μm are dispersed, is formed. The thickness of the cast strip is regulated to 0.01-1mm. By the above procedure, the cast slab can be prevent from being reduced to fine powder at the time of crushing for forming a magnet, and also the oxidation of the resulting powder can be prevented. By this method, the alloy excellent in magnetic properties can be obtained.

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CLAIMS

[Claim(s)]

[Claim 1] Fe60 - 88at% is used as a principal component B-2 - 15at% R10 - 25at%. The R2Fe14B mold arborescence of 3 micrometers - 15 micrometers of diameters of average minor-axis crystal grain or columnar crystal with which the diameter of minor-axis crystal grain contains a less than 1.0-micrometer fine crystal 10% or less, The cast piece for R-Fe-B system magnet alloys characterized by for the R-rich phase of 5 micrometers or less consisting of a homogeneity organization which distributed minutely, and cast piece thickness consisting of 0.01mm - 1.0mm.

[Claim 2] The magnet alloy molten metal which uses Fe60 - 88at% as a principal component B-2 - 15at% R10 - 25at% From temperature with a liquidus-line temperature [of an alloy] (coagulation initiation temperature) of +5 degrees C -

+300 degrees C With a quenching roll, with the primary cooling rate of 2x103 degree-C/sec-7x103 degree C/sec After cooling in cast piece temperature of 700 degrees C - 1000 degrees C, Said cast piece is cooled to the solidus-line temperature of an alloy after roll balking below (the completion temperature of coagulation) with the secondary cooling rate of 50 degrees C / min - 2x103 degrees C / min. The R2Fe14B mold arborescence of 3 micrometers - 15 micrometers of diameters of average minor-axis crystal grain or columnar crystal with which the diameter of minor-axis crystal grain contains a less than 1.0-micrometer fine crystal 10% or less, The manufacture approach of the cast piece for R-Fe-B system magnet alloys characterized by obtaining the cast piece for magnet alloys which R rich phase of 5 micrometers or less becomes from the homogeneity organization which distributed minutely, and cast piece thickness becomes from 0.01mm - 1.0mm.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the cast piece for R-Fe-B system

magnet alloys which has a detailed homogeneity organization, and its manufacture approach. After dissolving a R-Fe-B system alloy molten metal with a vacuum melting furnace, teeming is carried out to a quenching roll from the nozzle of a tundish point. By cooling the secondary cast piece which seceded from the molten metal from the roll after primary cooling with the specific cooling rate with a quenching roll with a specific cooling rate below to solidus-line temperature It is related with the cast piece for R-Fe-B system magnet alloys which obtains the quenching cast piece of the specific thickness which the R2Fe14B mold dendrite crystal or the columnar crystal which has the diameter of minor-axis crystal grain of a specific dimension, and specific R rich phase become from the homogeneity organization which distributed minutely, and its manufacture approach.

[0002]

[Description of the Prior Art] A magnet property high in the organization which has the main phase and R rich phase of a ternary system tetragonal compound is acquired, a R-Fe-B system permanent magnet (JP,59-46008,A) typical as a high performance permanent magnet is used in a field with the various electrical machinery products of ordinary homes to the broad peripheral device of a large-sized computer, and the R-Fe-B system permanent magnet of various presentations is proposed that it demonstrates the various magnet properties according to an application.

[0003] being 1 ferromagnetism phase and making [many] abundance of the R2Fe14 B phase of the main phase, in order to raise the residual magnetic flux density (Br) of a R-Fe-B system sintered magnet, raising the consistency of two sintered compacts to the theoretical density of the main phase, and 3 -- it is required further that the amount of preferred orientation of the direction of an easy axis of the main phase crystal grain should be raised.

[0004] Namely, although it is important for achievement of said 1st term to bring a magnetic presentation close to stoichiometric composition of the above-mentioned R2Fe14B If it is going to produce a R-Fe-B system sintered magnet

as a start raw material, the alloy lump which dissolved the alloy of the above-mentioned presentation and cast to mold From being [which was crystallized in the alloy lump] alpha-Fe, the R-rich phase being locally omnipresent, etc., grinding became difficult especially at the time of pulverizing, and there were problems, such as producing a presentation gap.

[0005] In order to prevent the residual of big-and-rough-izing of the fault slack crystal grain in the end of a R-Fe-B system alloy powder by the ingot grinding method, and alpha-Fe, and a segregation recently, the method of manufacturing a sintered magnet according to the usual powder-metallurgy processing is proposed [cast piece / the cast piece of specific board thickness, and / nothing and said cast piece] by the congruence rolling method in the R-Fe-B system alloy molten metal (JP,63-317643,A).

[0006] moreover, a R-Fe-B system alloy molten metal using a piece roll as an approach of manufacturing the quenching cast piece for permanent magnets by the horizontal **** strip cast method Prepare the nozzle of necessary width of face in the horizontal direction of a tundish point, make this nozzle adjoin, and support arrangement of the piece roll is carried out horizontally. After holding the molten metal dissolved with the high frequency fusion furnace in tundish, teeming of the molten metal is carried out to the piece roll side which level arrangement is carried out and carries out a continuation revolution from this nozzle, and the method of carrying out rapid solidification and manufacturing a quenching cast piece is proposed (JP,5-222488,A, JP,6-84624,A).

[0007] Furthermore, as a cast piece for magnet alloys which cast the R-Fe-B system magnet alloy molten metal in the quenching roll, R, T, and B are used as a principal component, the pitch diameter which consisted of R2Fe14 B phases substantially consists of a grain boundary phase which makes a subject the columnar grain which is 3-50 micrometers, and R rich phase, and the cast piece for magnet alloys whose thickness of the cooling direction is 0.1-2mm is proposed (JP,5-295490,A).

[0008]

[Problem(s) to be Solved by the Invention] Artificers did the knowledge of cast structure having changed with casting conditions a lot, and oxidation of the powder accompanying the pulverization at the time of grinding at the time of being magnet-ization and lowering of the amount of preferred orientation of a sintered magnet having taken place, and having had big effect on magnetic properties about the cast structure of the above-mentioned cast piece for magnet alloys, when it investigates in a detail.

[0009] In order that this invention may cancel the trouble in the cast piece for magnet alloys which cast the R-Fe-B system magnet alloy molten metal in the quenching roll, it can prevent oxidation of the powder accompanying the pulverization at the time of grinding in the case of magnet-izing, and lowering of the amount of preferred orientation of a sintered magnet, and aims at offer of the R-Fe-B system magnet alloy cast piece which clarified relation between a cast piece organization and magnetic properties so that the R-Fe-B system sintered magnet which was excellent in magnetic properties might be obtained, and offer of the manufacture approach of the magnet alloy cast piece concerned.

[0010]

[Means for Solving the Problem] The result to which artificers considered various relation of the magnetic properties of said cast piece organization for magnet alloys and sintered magnet, The arborescence or columnar crystal which has various magnitude and directions exists in said cast piece. Detailed arborescence or a detailed columnar crystal has big effect on oxidation of the powder accompanying the pulverization at the time of grinding in the case of magnet-izing, and lowering of the amount of preferred orientation of a sintered magnet. The knowledge of it being important reducing the detailed arborescence or columnar crystal in said cast piece is carried out. furthermore, when it inquires, in order to obtain the cast piece which reduced the detailed arborescence or columnar crystal in this cast piece After it carried out teeming of the alloy molten metal of specific temperature to the quenching roll from the nozzle and the 1st order cooled with the specific cooling rate, the knowledge of it being important

cooling the secondary cast piece which estranged the roll with a specific cooling rate below to solidus-line temperature was carried out, and this invention was completed.

[0011] Namely, this invention uses Fe60 - 88at% as a principal component B-2 - 15at% R10 - 25at%. The R2Fe14B mold arborescence of 3 micrometers - 15 micrometers of diameters of average minor-axis crystal grain or columnar crystal with which the diameter of minor-axis crystal grain contains a less than 1.0-micrometer fine crystal 10% or less, It is the cast piece for R-Fe-B system magnet alloys characterized by for the R-rich phase of 5 micrometers or less consisting of a homogeneity organization which distributed minutely, and cast piece thickness consisting of 0.01mm - 1.0mm.

[0012] This invention moreover, the magnet alloy molten metal which uses Fe60 - 88at% as a principal component B-2 - 15at% R10 - 25at% From temperature with a liquidus-line temperature [of an alloy] (coagulation initiation temperature) of +5 degrees C - +300 degrees C With a quenching roll, with the primary cooling rate of 2x103 degree-C/sec-7x103 degree C/sec After cooling in cast piece temperature of 700 degrees C - 1000 degrees C, Said cast piece is cooled to the solidus-line temperature of an alloy after roll balking below (the completion temperature of coagulation) with the secondary cooling rate of 50 degrees C / min - 2x103 degrees C / min. The R2Fe14B mold arborescence of 3 micrometers - 15 micrometers of diameters of average minor-axis crystal grain or columnar crystal with which the diameter of minor-axis crystal grain contains a less than 1.0-micrometer fine crystal 10% or less, It is the manufacture approach of the cast piece for R-Fe-B system magnet alloys characterized by obtaining the cast piece for magnet alloys which R rich phase of 5 micrometers or less becomes from the homogeneity organization which distributed minutely, and cast piece thickness becomes from 0.01mm - 1.0mm.

[0013]

[Function] After this invention dissolves a R-Fe-B system alloy molten metal with a vacuum melting furnace, By carrying out teeming to a quenching roll from the

nozzle of a tundish point, and cooling the secondary cast piece which seceded from the molten metal from the roll after primary cooling to specific temperature with the specific cooling rate with a quenching roll with a specific cooling rate below to solidus-line temperature It is characterized by obtaining the quenching cast piece of the specific thickness which the R2Fe14B mold dendrite crystal or the columnar crystal which has the diameter of minor-axis crystal grain of a specific dimension, and specific R rich phase become from the homogeneity organization which distributed minutely.

[0014] That is, board thickness became thin, and although cast structure of a cooling cast piece was made detailed, it found out that cast structure changed with the temperature of the cast piece in the event of leaving a quenching roll actually, and a subsequent cooling rate, so that it was decided that it would be the flash when the molten metal contacted the cooling roller, the contact length of a molten metal and a cooling roller was short and roll peripheral speed was quick. Generally, coagulation begins at liquidus-line temperature and coagulation completes an alloy molten metal at solidus-line temperature. However, if the time amount which passes through the solid-liquid coexistence field from this liquidus line to solidus-line temperature is long, cast structure will make it big and rough. With a R-Fe-B system alloy, since the difference of said liquidus-line temperature and solidus-line temperature is as large as about 500 degrees C, especially the aforementioned big-and-rough-izing is remarkable.

[0015] That is, the cast piece temperature immediately after estranging a quenching roll will grow, if a subsequent cooling rate is slow and the time amount which passes through a solid-liquid coexistence field becomes long, although a detailed organization will be obtained if subsequent cooling of more than the solidus line is quick enough, and crystal grain invites lowering of iHc of a sintered magnet. As a result of artificers' investigating the relation of said pass time and diameter of crystal grain, the diameter of crystal grain grows [the pass time of a solid-liquid coexistence field] even several small minutes, for example, when the pass time from 800 degrees C to solidus-line temperature is 3 minutes, the

diameter of crystal grain grows up into 20-30 micrometers.
[0016] Moreover, although cooling with a roll can be strengthened, the cast piece at the time of roll balking can be made below into solidus-line temperature, and big and rough-ization of said crystal grain does not take place in this case, the rate of cooling with a roll is too quick, and a crystal is made detailed too much and invites lowering of Br of a sintered magnet. That is, the knowledge of the two-step cooling method which cools the primary alloy molten metal to specific temperature with a specific cooling rate with a quenching roll in order not to make the diameter of crystal grain of a cast piece make it detailed too much, and cools further the secondary cast piece from which it seceded from the quenching roll after that with a specific cooling rate below to solidus-line temperature in order not to make the detailed organization make it big and rough being important was carried out.
[0017] The reason which limited the temperature of the alloy molten metal which carries out cooling coagulation with a quenching roll in the manufacture approach of the cast piece this invention to the liquidus-line temperature (coagulation initiation temperature) of +5 degrees C - +300 degrees C Since it becomes impossible to cast, if an alloy molten metal solidifies in the nozzle section, and it causes nozzle plugging at the liquidus-line temperature of less than +5 degrees C, and it is not desirable and the liquidus-line temperature of +300 degrees C is exceeded Since molten metal temperature is too high, the molten metal temperature which cooling with a roll becomes inadequate, and the diameter of average minor-axis crystal grain exceeds 15 micrometers, and contacts a roll is high and the life of a cooling roller becomes short, it is not desirable.
[0018] Setting to this invention, a primary cooling rate is {(molten metal temperature which carries out roll contact) -(cast piece temperature at time of roll balking)}/(roll contact time).
It is alike, and defines, under 2x103 degrees C / sec of cooling of the molten metal according [a primary cooling rate] to a roll are inadequate, and the diameter of average minor-axis crystal grain is not desirable more than 15

micrometers, and if 7x103 degrees C/sec is exceeded, since a fine crystal with a particle size of 1 micrometer or less exceeds [the diameter of average minor-axis crystal grain] 10% at least 3 micrometers or more by average minor-axis crystal grain liquid becoming detailed with less than 3 micrometers, it is not desirable. Moreover, the range where a primary cooling rate is desirable is 3x103 degree-C/sec-6x103 degree C/sec.

[0019] The reason which limited the cast piece temperature after primary cooling to 700 degrees C - 1000 degrees C At less than 700 degrees C, the diameter of average minor-axis crystal grain becomes detailed with less than 3 micrometers, and if the diameter of average minor-axis crystal grain exceeds 1000 degrees C further preferably in order that a fine crystal 1 micrometer or less may exceed 10% at least 3 micrometers or more Since the secondary cooling system with which an installation cost increases is needed in order for the time amount cooled after roll balking of a cast piece and to below solidus-line temperature to become long, and for the diameter of average minor-axis crystal grain to make it big and rough exceeding 15 micrometers and to cool below to solidus-line temperature for a short time, it is not desirable. Furthermore, the cast piece temperature requirement after desirable primary cooling is 700 degrees C - 900 degrees C.

[0020] the solid-liquid coexistence field in which the reason which limited cooling of the cast piece after roll balking to below solidus-line temperature exceeded solidus-line temperature in this invention -- R -- the rich liquid phase exists, and since a crystal grows, even maintenance for several small minutes makes big and rough and a magnet property, especially coercive force reduce, a crystal does not grow, namely, needs to cool below to the solidus-line temperature in which the liquid phase does not exist at all.

[0021] Setting to this invention, a secondary cooling rate is {(at time of roll balking cast piece temperature) -(solidus-line temperature)}/(cooldown delay).

It is not desirable, in order that it is alike, it may define, the time amount from which passage takes a solid-liquid coexistence field to a secondary cooling rate

at under 50 degrees C / min may become long and a crystal may grow and make it big and rough. Moreover, although it is desirable, in consideration of facility cost etc., less than 2x103 degrees C / min of a mass production target are more desirable [the time amount which passage of a solid-liquid coexistence field takes becomes short and], as a secondary cooling rate is quick. Moreover, the range where a secondary cooling rate is desirable is 100 to 2x103 degrees C / min.

[0022] It can cool in migration, or between a quenching roll and a cast piece hold box, by inert gas cooling, a conveyor, or belts, such as Ar gas, further, within a cast piece hold box, inert gas cooling can be carried out and secondary cooling in this invention can be adjusted, and on both sides of a cast piece, it may cool with two pairs of rotating belts, or it may have the approach of supplying to Liquid Ar directly etc., and the combination of these approaches is sufficient as it. Moreover, in order to realize sufficient secondary cooling rate, it is necessary to take enough the distance between a cooling roller and a cast piece hold box, and, as for the distance, 1/20 or more [of roll peripheral velocity] is desirable. For example, when roll peripheral velocity is 100 m/min, it is 5m or more.

[0023] In the cast piece for magnet alloys of this invention, the diameter of minor-axis crystal grain means the die length of the minor axis of a vertical direction to arborescence or the direction of a major axis of a columnar crystal. The reason which limited the R2Fe14B mold arborescence of the cast piece for magnet alloys, or the diameter of average minor-axis crystal grain of a columnar crystal to 3 micrometers - 15 micrometers When disintegration is carried out in less than 3 micrometers, become easy to oxidize, and degradation of magnetic properties is invited. Moreover, since the end of an alloy powder disintegration was carried out becomes the polycrystalline substance, the amount of preferred orientation at the time of press forming invites turbulence and lowering of magnetic Br, the diameter of crystal grain of a sintered magnet will become large if it exceeds 15 micrometers further, and coercive force declines, it is not desirable.

[0024] Moreover, the rate of the polycrystalline substance in the end of an alloy

powder disintegration was carried out in content exceeding 10% increases, and since Br of turbulence and a magnet falls, the amount of preferred orientation at the time of press forming is not desirable [the reason to which the diameter of minor-axis crystal grain limited content of a less than 1.0-micrometer fine crystal to 10% or less].

[0025] each rate of a quantitative ratio of the R2Fe14B mold dendrite crystal in the homogeneity organization which distributed minutely [the cast piece for magnet alloys of this invention], a columnar crystal, and R rich phase -- R2Fe14B mold dendrite crystal or a columnar crystal -- 90% or more -- desirable -- further -- desirable -- 95% or more -- it is -- moreover, R rich phase -- 3 - 10% - it is desirable. In this invention, although changed, when [which depends solidus-line temperature on a R-Fe-B system magnet presentation] a magnet presentation is a 14Nd-79Fe-7Bat% magnet, solidus-line temperature is 665 degrees C.

[0026] The reason for definition of an alloy presentation of the alloy cast piece which manufactures the R-Fe-B system permanent magnet by this invention is explained below. The rare earth elements R contained in the alloy cast piece for permanent magnets of this invention are rare earth elements which include an yttrium (Y) and include light rare earth and heavy rare earth. As R, it is sufficient with light rare earth, and especially Nd and Pr are desirable. Moreover, it has one sort in R, and although it is sufficient, two or more kinds of mixture (a misch metal, didym, etc.) can be practically used for the reasons of the facilities on acquisition etc., and Sm, Y, La, Ce, Gd, etc. can usually be used as mixture with other R, especially Nd, Pr, etc. In addition, this R may not be pure rare earth elements, and what contains an unescapable impurity on manufacture in the available range on industry does not interfere.

[0027] R will be the essential element of the alloy cast piece which manufactures a R-Fe-B system permanent magnet, under by 10 atom %, high magnetic properties, especially high coercive force are not acquired, if 25 atom % is exceeded, a residual magnetic flux density (Br) will fall and the permanent

magnet of the outstanding property will not be obtained. Therefore, R is taken as the range of ten atoms % - 25 atom %.

[0028] B is the essential element of the alloy cast piece which manufactures a R-Fe-B system permanent magnet, and under by 2 atom %, high coercive force (iHc) is not acquired, and since a residual magnetic flux density (Br) will fall if an atom is exceeded 15%, the outstanding permanent magnet is not obtained. Therefore, B is taken as the range of two atoms % - 15 atom %.

[0029] Since high coercive force will not be acquired if Fe is the essential element of the alloy cast piece which manufactures a R-Fe-B system permanent magnet, a residual magnetic flux density (Br) falls under by 60 atom % and an atom is exceeded 88%, Fe is limited to 60 atoms % - 88 atom %. Moreover, although it is replaceable in a part of Fe at one sort of Co and nickel, or two sorts and this is because the effectiveness of raising the effectiveness of raising the temperature characteristic of a permanent magnet, and corrosion resistance is acquired, if one sort of Co and nickel or two sorts exceed 50% of Fe, high coercive force will not be acquired and the outstanding permanent magnet will not be obtained. Therefore, one sort or two sorts of amounts of permutations, Co and nickel, make 50% of Fe an upper limit.

[0030] In the alloy cast piece by this invention, in order to obtain the outstanding permanent magnet which has both a high residual magnetic flux density and high coercive force, R12 atom % - 16 atom %, B4 atom % - 12 atom %, and Fe72 atom % - 84 atom % are desirable. Moreover, although the alloy cast piece by this invention can permit existence of unescapable-on industrial production impurities, such as oxygen besides R, B, and Fe, and C, calcium, Mg The manufacturability improvement of a magnet alloy and low-pricing are possible by permuting a part of B below by 4.0 atom % with at least one sort and the total quantity among Cu(s) below C below 4.0 atom %, P below 3.5 atom %, S below 2.5 atom %, and 3.5 atom %. The corrosion resistance of a sintered magnet improves by permuting said a part of B by C below 4.0 atom % especially.

[0031] Into furthermore, the R-Fe-B alloy containing said R and B, Fe alloy, or Co

aluminum below 9.5 atom %, Ti below 4.5 atom %, V below 9.5 atom %, Cr below 8.5 atom %, Mn below 8.0 atom %, Bi below pentatomic %, Nb below 12.5 atom %, Ta below 10.5 atom %, Mo below 9.5 atom %, The high coercive force of a permanent magnet alloy becomes possible by carrying out at least one-sort addition content among W below 9.5 atom %, Sb below 2.5 atom %, germanium below 7 atom %, Sn below 35 atom %, Zr below 5.5 atom %, and Hf below 5.5 atom %. In the R-Fe-B system permanent magnet of this invention, as for a crystal phase, it is indispensable that the main phase is ******, and it is effective for obtaining the detailed and uniform end of an alloy powder, and producing especially, the sintering permanent magnet which has outstanding magnetic properties.

[0032] The reason which limited the board thickness of the magnet alloy cast piece which has the homogeneity organization which arborescence or the columnar crystal, and the R-rich phase distributed minutely in this invention to 0.01mm - 10mm In less than 0.01mm, a large next door and the diameter of crystal grain serve as smallness from 3 micrometers, and since it becomes easy to oxidize when the quenching effectiveness carries out disintegration, while it invites degradation of magnetic properties Since the particle after pulverizing serves as polycrystal, the amount of preferred orientation falls and Br falls, if it is not desirable and 10mm is exceeded, since a cooling rate will become slow, it will be easy to crystallize alpha-Fe and the diameter of crystal grain will also produce omnipresence of a large next door and Nd rich phase, Since magnetic properties, especially coercive force decline, it is because it is not desirable. It is 0.05mm - 0.8mm of board thickness more preferably.

[0033] Although or more about 1 / 10 are detailed compared with the thing of the ingot which the $R_2Fe_{14}B$ crystal of the main phase cast the cross-section organization of the R-Fe-B system alloy of the specific presentation acquired by the strip casting method of this invention to the conventional mold, and was obtained, the diameter of average minor-axis crystal grain in which the diameter of minor-axis crystal grain contains a less than 1.0-micrometer fine crystal 10%

or less is 3 micrometers - 15 micrometers like the above-mentioned.
[0034]
[Example]
In an example 1Ar reduced pressure 200torr ambient atmosphere, the alloy molten metal with a molten metal temperature of 1300 degrees C of a 31Nd-1.0Dy-1.1B-3.0Co-** Fe (wt%) presentation (liquidus-line temperature of 1170 degrees C) From a nozzle, to a water-cooled Cu piece roll surface with an outer diameter [of rotational frequency 120rpm] of 300 micrometers In the primary cooling rate of 5x103 degrees C / sec, after cooling in cast piece temperature of 800 degrees C, Ar gas of the pressure of 5kg/cm2 and flow rate 500 l/min is sprayed from the upper and lower sides of a cast piece between a quenching roll and a cast piece hold box (distance of 8m) after roll balking. Furthermore, Ar gas of the pressure of 5kg/cm2, and the flow rate of 500l. / min was sprayed within the cast piece hold box, the gas cooling method of the cast piece was carried out with the secondary cooling rate of 200 degrees C / min to 600 degrees C (solidus-line temperature of 660 degrees C), and the cast piece with a thickness of 0.38mm was obtained.
[0035] Mirror plane polishing of the cross section of the obtained cast piece was carried out, and it observed with the optical microscope (one 400 times the scale factor of this), and as a result of measuring the diameter of minor-axis crystal grain in linear analysis about 500 crystals, it had the homogeneity organization which the R2Fe14B mold dendrite crystal of 4.5 micrometers of diameters of average minor-axis crystal grain of 3% content of a fine crystal 1.0 micrometers or less of the diameter of minor-axis crystal grain and the R-rich phase of 5 micrometers or less distributed minutely. The obtained cast piece was pulverized by jet mill grinding after coarse grinding, and impalpable powder with an average powder particle size of 3.0 micrometers was obtained. The magnetic properties and the diameter of average crystal grain of a test piece which were obtained after molding in magnetic-field-intensity 15kOe at press ** 1 ton/cm2 by performing the 4-hour sintering back at 1040 degrees C, and performing aging

treatment of 1 hour at 600 degrees C in a vacuum are shown for said powder in a table 1.
[0036] Using the alloy molten metal of the same presentation as example of comparison 1 example 1, the same roll as an example 1 was used, it cooled in the primary cooling rate of 7500 degrees C / sec, and the cast piece temperature at the time of roll balking was 630 degrees C. Furthermore, the gas cooling method of the cast piece after roll balking was carried out with the secondary cooling rate of 200 degrees C / min, and the cast piece of 0.30mm of cast piece thickness was obtained. As a result of measuring the diameter of minor-axis crystal grain by the same approach as the example 1 of the obtained cast piece, the R2Fe14B mold dendrite crystal of 3.2 micrometers of diameters of average minor-axis crystal grain of 18% content of the fine crystal of 1 micrometer or less of diameters of minor-axis crystal grain was obtained. The sintered magnet was obtained on the same conditions as an example 1 except pulverizing the obtained cast piece in average powder particle size of 2.9 micrometers. Magnetic properties and the measurement result of the diameter of average crystal grain are shown in a table 1.
[0037] Using the alloy molten metal of the same presentation as example of comparison 2 example 1, the same roll as an example 1 was used, it cooled by the primary cooling rate of 1600 degrees C / sec, and cast piece temperature was 1100 degrees C. Furthermore, the gas cooling method of the cast piece after roll balking was carried out with the secondary cooling rate of 100 degrees C / min to 600 degrees C, and the cast piece of 0.43mm of cast piece thickness was obtained. Although the fine crystal of 1 micrometer or less of diameters of minor-axis crystal grain was 0% as a result of measuring the diameter of minor-axis crystal grain by the same approach as an example 1, the diameter of average minor-axis crystal grain was 32 micrometers. The sintered magnet was obtained on the same conditions as an example 1 except pulverizing the obtained cast piece in average powder particle size of 3.2 micrometers. Magnetic properties and the measurement result of the diameter of average crystal grain are shown

in a table 1.

[0038] Using the alloy molten metal of the same presentation as example of comparison 3 example 1, the same roll as an example 1 was used and the cast piece of 0.38 micrometers of cast piece thickness was obtained on the same manufacture conditions as an example 1 except setting a secondary cooling rate to 20 degrees C / min. Although the fine crystal of 1 micrometer or less of diameters of minor-axis crystal grain was 0.5% as a result of measuring the diameter of minor-axis crystal grain by the same approach as an example 1, it was 21 micrometers of diameters of average minor-axis crystal grain. The sintered magnet was obtained on the same conditions as an example 1 except pulverizing the obtained cast piece in average powder particle size of 3.4 micrometers. The magnetic properties of a sintered magnet and the measurement result of the diameter of average crystal grain are shown in a table 1.

[0039] After using the alloy molten metal of the same presentation as example of comparison 4 example 1, and the same roll and carrying out a gas cooling method to 750 degrees C by the secondary cooling rate of 250 degrees C / min, the cast piece of 0.39 micrometers of cast piece thickness was obtained on the same manufacture conditions as an example 1 except cooling by 20 degrees C / min to 600 degrees C. Although the fine crystal of 1 micrometer or less of diameters of minor-axis crystal grain was 0.8% as a result of measuring the diameter of minor-axis crystal grain by the same approach as an example 1, average minor-axis particle size was 18 micrometers. The sintered magnet was obtained on the same conditions as an example 1 except pulverizing the obtained cast piece in average powder particle size of 3.3 micrometers. The magnetic properties of the obtained sintered magnet and the measurement result of the diameter of average crystal grain are shown in a table 1.

[0040]

[A table 1]

	焼結磁石の磁気特性			焼結磁石の平均結晶粒径
	Br (kG)	(BH)max (MGOe)	iHc (kOe)	
実施例1	13.26	42.6	16.4	6.2μm
比較例1	12.80	39.7	16.8	6.3μm
比較例2	13.26	42.6	13.4	12.8μm
比較例3	13.24	42.5	13.7	10.1μm
比較例4	13.22	42.4	14.2	9.6μm

[0041]

[Effect of the Invention] After this invention dissolves a R-Fe-B system alloy molten metal with a vacuum melting furnace, By carrying out teeming to a quenching roll from the nozzle of a tundish point, and cooling the secondary cast piece which seceded from the molten metal from the roll after primary cooling with the specific cooling rate with a quenching roll with a specific cooling rate below to solidus-line temperature It is what obtains the quenching cast piece of the specific thickness which the R2Fe14B mold dendrite crystal or the columnar crystal which has the diameter of minor-axis crystal grain of a specific dimension, and specific R rich phase become from the homogeneity organization which distributed minutely. The pulverization at the time of lowering of the amount of preferred orientation and grinding in the case of magnet-izing and powdered oxidation can be prevented, and the R-Fe-B system magnet alloy cast piece which was excellent in magnetic properties is obtained.

[Translation done.]

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(54)【発明の名称】　Ｒ－Ｆｅ－Ｂ系磁石合金用鋳片及びその製造方法

(57)【要約】

【目的】　Ｒ－Ｆｅ－Ｂ系磁石合金溶湯を急冷ロールにて鋳造した磁石合金用鋳片における問題点を解消するため、配向度の低下及び磁石化の際の粉砕時の微粉化、粉末の酸化を防止でき、磁気特性の優れたＲ－Ｆｅ－Ｂ系磁石合金鋳片が得られるように鋳片組織と磁気特性の関係を明確にしたＲ－Ｆｅ－Ｂ系磁石合金鋳片とその製造方法の提供。

【構成】　Ｒ－Ｆｅ－Ｂ系合金溶湯を真空溶解炉にて溶解した後、タンディシュ先端部のノズルより急冷ロールに注湯し、溶湯を急冷ロールにて特定の冷却速度にて１次冷却後、ロールより離脱した鋳片を固相線温度以下に特定の冷却速度にて２次冷却することにより、特定寸法の短軸結晶粒径を有する$R_2Fe_{14}B$型樹枝状結晶あるいは柱状結晶と特定のＲリッチ相とが微細に分散した均質組織からなる特定厚の急冷鋳片を得るもので、配向度の低下及び磁石化の際の粉砕時の微粉化、粉末の酸化を防止でき、磁気特性の優れたＲ－Ｆｅ－Ｂ系磁石合金鋳片が得られる。

【特許請求の範囲】

【請求項1】　R10～25at%、B2～15at%、Fe60～88at%を主成分とし、短軸結晶粒径が1．0μm未満の微細結晶を10%以下含有する平均短軸結晶粒径3μm～15μmの$R_2Fe_{14}B$型樹枝状あるいは柱状結晶と、5μm以下のR－リッチ相とが、微細に分散した均質組織からなり、鋳片厚みが0．01mm～1．0mmからなることを特徴とするR－Fe－B系磁石合金用鋳片。

【請求項2】　R10～25at%、B2～15at%、Fe60～88at%を主成分とする磁石合金溶湯を、合金の液相線温度（凝固開始温度）＋5℃～＋300℃の温度より、急冷ロールにて2×10^3℃／sec～7×10^3℃／secの1次冷却速度にて鋳片温度700℃～1000℃に冷却後、ロール離脱後に前記鋳片を合金の固相線温度に（凝固完了温度）以下に50℃／min～2×10^3℃／minの2次冷却速度にて冷却し、短軸結晶粒径が1．0μm未満の微細結晶を10%以下含有する平均短軸結晶粒径3μm～15μmの$R_2Fe_{14}B$型樹枝状あるいは柱状結晶と、5μm以下のRリッチ相とが、微細に分散した均質組織からなり、鋳片厚みが0．01mm～1．0mmからなる磁石合金用鋳片を得ることを特徴とするR－Fe－B系磁石合金用鋳片の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】この発明は、微細均質組織を有するR－Fe－B系磁石合金用鋳片及びその製造方法に係り、R－Fe－B系合金溶湯を真空溶解炉にて溶解した後、タンディシュ先端部のノズルより急冷ロールに注湯し、溶湯を急冷ロールにて特定の冷却速度にて1次冷却後、ロールより離脱した鋳片を固相線温度以下に特定の冷却速度にて2次冷却することにより、特定寸法の短軸結晶粒径を有する$R_2Fe_{14}B$型樹枝状結晶あるいは柱状結晶と特定のRリッチ相とが微細に分散した均質組織からなる特定厚の急冷鋳片を得るR－Fe－B系磁石合金用鋳片及びその製造方法に関する。

【0002】

【従来の技術】高性能永久磁石として代表的なR－Fe－B系永久磁石（特開昭59－46008号）は、三元系正方晶化合物の主相とRリッチ相を有する組織にて高い磁石特性が得られ、一般家庭の各種電器製品から大型コンピュータの周辺機器まで幅広い分野で使用され、用途に応じた種々の磁石特性を発揮するよう種々の組成のR－Fe－B系永久磁石が提案されている。

【0003】R－Fe－B系焼結磁石の残留磁束密度（Br）を高めるためには、1）強磁性相であり、主相の$R_2Fe_{14}B$相の存在量を多くすること、2）焼結体の密度を主相の理論密度まで高めること、3）さらに、主相結晶粒の磁化容易軸方向の配向度を高めることが要求される。

【0004】すなわち、前記1）項の達成のためには、磁石の組成を上記$R_2Fe_{14}B$の化学量論的組成に近づけることが重要であるが、上記組成の合金を溶解し、鋳型に鋳造した合金塊を、出発原料としてR－Fe－B系焼結磁石を作製しようとすると、合金塊に晶出したα－Feや、R－rich相が局部的に遍在していることなどから、特に微粉砕時に粉砕が困難となり、組成ずれを生ずる等の問題があった。

【0005】最近、鋳塊粉砕法によるR－Fe－B系合金粉末の欠点たる結晶粒の粗大化、α－Feの残留、偏析を防止するために、R－Fe－B系合金溶湯を双ロール法により、特定板厚の鋳片となし、前記鋳片を通常の粉末冶金法に従って、焼結磁石を製造する方法が提案（特開昭63－317643号公報）されている。

【0006】また、R－Fe－B系合金溶湯を片ロールを用いて、横注ぎストリップキャスト法により永久磁石用急冷鋳片を製造する方法として、タンディッシュ先端部の水平方向に所要幅のノズルを設け、このノズルに隣接させて片ロールを水平方向に軸支配置し、高周波溶解炉にて溶解した溶湯をタンディッシュに収容後、該ノズルから溶湯を水平配置されて連続回転する片ロール面に注湯して、急冷凝固させて急冷鋳片を製造する方法が提案（特開平5－222488号公報、特開平6－84624号公報）されている。

【0007】さらに、R－Fe－B系磁石合金溶湯を急冷ロールにて鋳造した磁石合金用鋳片として、R、T、及びBを主成分とし、実質的に$R_2Fe_{14}B$相から構成された平均径が3～50μmの柱状結晶粒とRリッチ相を主体とする結晶粒界相からなり、冷却方向の厚さが0．1～2mmである磁石合金用鋳片が提案（特開平5－295490号公報）されている。

【0008】

【発明が解決しようとする課題】発明者らは、上記磁石合金用鋳片の鋳造組織について、詳細に調査したところ、鋳造条件により鋳造組織が大きく変化し、磁石化の際の粉砕時の微粉化に伴う粉末の酸化、および焼結磁石の配向度の低下が起こり、磁気特性に大きな影響を及ぼしていることを知見した。

【0009】この発明は、R－Fe－B系磁石合金溶湯を急冷ロールにて鋳造した磁石合金用鋳片における問題点を解消するため、磁石化の際の粉砕時の微粉化に伴う粉末の酸化および焼結磁石の配向度の低下を防止でき、磁気特性の優れたR－Fe－B系焼結磁石が得られるように鋳片組織と磁気特性の関係を明確にしたR－Fe－B系磁石合金鋳片の提供と、当該磁石合金鋳片の製造方法の提供を目的としている。

【0010】

【課題を解決するための手段】発明者らは、前記磁石合金用鋳片組織と焼結磁石の磁気特性の関係を種々検討し

た結果、前記鋳片には種々の大きさや方向を有する樹枝状もしくは柱状結晶が存在し、微細な樹枝状もしくは柱状結晶が、磁石化の際の粉砕時の微粉化に伴う粉末の酸化および焼結磁石の配向度の低下に大きな影響を及ぼし、前記鋳片内の微細樹枝状もしくは柱状結晶を低減することが重要であることを知見し、更に検討したところ、かかる鋳片内の微細樹枝状もしくは柱状結晶を低減した鋳片を得るためには、特定温度の合金溶湯をノズルより急冷ロールに注湯して、特定の冷却速度にて1次冷却した後、ロールを離間した鋳片を固相線温度以下に特定の冷却速度にて2次冷却することが重要であることを知見し、この発明を完成した。

【0011】すなわち、この発明は、R10～25at%、B2～15at%、Fe60～88at%を主成分とし、短軸結晶粒径が1．0μm未満の微細結晶を10%以下含有する平均短軸結晶粒径3μm～15μmの$R_2Fe_{14}B$型樹枝状あるいは柱状結晶と、5μm以下のR－リッチ相とが、微細に分散した均質組織からなり、鋳片厚みが0．01mm～1．0mmからなることを特徴とするR－Fe－B系磁石合金用鋳片である。

【0012】また、この発明は、R10～25at%、B2～15at%、Fe60～88at%を主成分とする磁石合金溶湯を、合金の液相線温度（凝固開始温度）＋5℃～＋300℃の温度より、急冷ロールにて2×10^3℃／sec～7×10^3℃／secの1次冷却速度にて鋳片温度700℃～1000℃に冷却後、ロール離脱後に前記鋳片を合金の固相線温度に（凝固完了温度）以下に50℃／min～2×10^3℃／minの2次冷却速度にて冷却し、短軸結晶粒径が1．0μm未満の微細結晶を10%以下含有する平均短軸結晶粒径3μm～15μmの$R_2Fe_{14}B$型樹枝状あるいは柱状結晶と、5μm以下のRリッチ相とが、微細に分散した均質組織からなり、鋳片厚みが0．01mm～1．0mmからなる磁石合金用鋳片を得ることを特徴とするR－Fe－B系磁石合金用鋳片の製造方法である。

【0013】

【作用】この発明は、R－Fe－B系合金溶湯を真空溶解炉にて溶解した後、タンディシュ先端部のノズルより急冷ロールに注湯し、溶湯を急冷ロールにて特定の冷却速度で特定の温度まで1次冷却後、ロールより離脱した鋳片を固相線温度以下に特定の冷却速度にて2次冷却することにより、特定寸法の短軸結晶粒径を有する$R_2Fe_{14}B$型樹枝状結晶あるいは柱状結晶と特定のRリッチ相とが微細に分散した均質組織からなる特定厚の急冷鋳片を得ることを特徴とする。

【0014】すなわち、冷却鋳片の鋳造組織は、溶湯が冷却ロールに接触した瞬間に決定され、溶湯と冷却ロールの接触長が短く、ロール周速が速い程、板厚は薄くなり微細化されるが、現実には急冷ロールを離れる時点での鋳片の温度およびその後の冷却速度によって、鋳造組織が変化することを見出した。一般に合金溶湯は液相線温度で凝固が開始し、固相線温度で凝固が完了する。しかし、この液相線から固相線温度までの固液共存領域を通過する時間が長いと鋳造組織は粗大化する。R－Fe－B系合金では前記液相線温度と固相線温度の差が約500℃と大きいため、特に前記粗大化は顕著である。

【0015】すなわち、急冷ロールを離間した直後の鋳片温度が固相線以上でも、その後の冷却が十分速ければ微細組織が得られるが、その後の冷却速度が遅く、固液共存領域を通過する時間が長くなると、結晶粒は成長し、焼結磁石のiHcの低下を招来する。発明者らが前記通過時間と結晶粒径の関係を調べた結果、固液共存領域の通過時間が僅か数分でも結晶粒径が成長し、例えば800℃から固相線温度までの通過時間が3分の場合、結晶粒径は20～30μmに成長する。

【0016】またロールでの冷却を強化して、ロール離脱時の鋳片を固相線温度以下にすることができるが、この場合、前記結晶粒の粗大化は起こらないが、ロールによる冷却の速度が速すぎ結晶が微細化されすぎて、焼結磁石のBrの低下を招来する。すなわち、鋳片の結晶粒径を微細化させすぎないためには、合金溶湯を急冷ロールにて特定の冷却速度で特定の温度まで1次冷却し、さらにその後、急冷ロールより離脱した鋳片をその微細組織を粗大化させないためには固相線温度以下に特定の冷却速度で2次冷却する2段階冷却法が重要であることを知見したのである。

【0017】この発明の鋳片の製造方法において、急冷ロールにて冷却凝固する合金溶湯の温度を液相線温度（凝固開始温度）＋5℃～＋300℃に限定した理由は、液相線温度＋5℃未満ではノズル部で合金溶湯が凝固して、ノズルづまりを起こし、鋳造できなくなるので好ましくなく、また、液相線温度＋300℃を越えると、溶湯温度が高すぎて、ロールでの冷却が不十分となり、平均短軸結晶粒径が15μmを越え、また、ロールに接触する溶湯温度が高いため、冷却ロールの寿命が短くなるので、好ましくない。

【0018】この発明において、1次冷却速度は
｛（ロール接触する溶湯温度）－（ロール離脱時の鋳片温度）｝／（ロール接触時間）
にて定義され、1次冷却速度が2×10^3℃／sec未満ではロールによる溶湯の冷却が不十分で、平均短軸結晶粒径が15μmを越えて好ましくなく、また、7×10^3℃／secを越えると、平均短軸結晶粒液が3μm未満と微細になり、また平均短軸結晶粒径が3μm以上でも、粒径1μm以下の微細結晶が10%を越えるので好ましくない。また、1次冷却速度の好ましい範囲は、3×10^3℃／sec～6×10^3℃／secである。

【0019】1次冷却後の鋳片温度を700℃～1000℃に限定した理由は、700℃未満では平均短軸結晶粒径が3μm未満と微細になり、また、平均短軸結晶粒

径が3μm以上でも、1μm以下の微細結晶が10％を越えるため好ましくなく、さらに、1000℃を超えると、鋳片のロール離脱後、固相線温度以下まで冷却する時間が長くなり平均短軸結晶粒径が15μmを超えて、粗大化し、又固相線温度以下に短時間に冷却するためには設備費のかさむ2次冷却装置が必要となるので、好ましくない。更に、好ましい1次冷却後の鋳片温度範囲は、700℃～900℃である。

【0020】この発明において、ロール離脱後の鋳片の冷却を固相線温度以下に限定した理由は、固相線温度を超えた固液共存領域では、Rリッチな液相が存在し、僅か数分の保持でも結晶が成長し粗大化して、磁石特性、特に保磁力を低下させるので、結晶が成長しない、すなわち、液相が全く存在しない固相線温度以下まで冷却する必要がある。

【0021】この発明において、2次冷却速度は、

｛（ロール離脱時鋳片温度）－（固相線温度）｝／（冷却時間）

にて定義づけられ、2次冷却速度が50℃／min未満では固液共存領域を通過に要する時間が長くなり、結晶が成長し粗大化するため好ましくない。また、2次冷却速度は速ければ速い程、固液共存領域の通過に要する時間が短くなり好ましいが、量産的は設備コスト等を考慮して、2×10^3℃／min以内が好ましい。また、2次冷却速度の好ましい範囲は、100～2×10^3℃／minである。

【0022】この発明における2次冷却は、急冷ロールと鋳片収容箱間にてArガス等の不活性ガス冷却、あるいはコンベア又はベルトにて移送中にて冷却したり、更に鋳片収容箱内にて不活性ガス冷却して調節することができ、また、2対の回転するベルトによって、鋳片を挟んで冷却したり、液体Arに直接投入する方法などがあり、これらの方法の組合せでもよい。また、充分な2次冷却速度を実現するためには、冷却ロールと鋳片収容箱間の距離を十分とる必要があり、その距離はロール周速度の1／20以上が好ましい。例えば、ロール周速度が100m／minの場合は5m以上である。

【0023】この発明の磁石合金用鋳片において、短軸結晶粒径は樹枝状もしくは柱状結晶の長軸方向に対して垂直な方向の短軸の長さを意味する。磁石合金用鋳片の$R_2Fe_{14}B$型樹枝状もしくは柱状結晶の平均短軸結晶粒径を3μm～15μmに限定した理由は、3μm未満では粉末化した時に酸化しやすくなり、磁気特性の劣化を招来し、また粉末化した合金粉末が多結晶体となり、プレス成形時の配向度が乱れ、磁石のBrの低下を招来し、さらに、15μmを超えると焼結磁石の結晶粒径が大きくなり、保磁力が低下するため、好ましくない。

【0024】また、短軸結晶粒径が1.0μm未満の微細結晶の含有を10％以下に限定した理由は、10％を越える含有では粉末化した合金粉末中の多結晶体の割合が増加し、プレス成形時の配向度が乱れ、磁石のBrが低下するので好ましくない。

【0025】この発明の磁石合金用鋳片の微細に分散した均質組織における、$R_2Fe_{14}B$型樹枝状結晶、柱状結晶、Rリッチ相の各量比率は、$R_2Fe_{14}B$型樹枝状結晶もしくは柱状結晶は90％以上が好ましく、更に好ましくは95％以上であり、又Rリッチ相は3～10％が・好ましい。この発明において、固相線温度はR－Fe－B系磁石組成による変動するが、磁石組成が14Nd－79Fe－7Bat％磁石の場合は、固相線温度は665℃である。

【0026】以下にこの発明によるR－Fe－B系永久磁石を製造する合金鋳片の合金組成の限定理由を説明する。この発明の永久磁石用合金鋳片に含有される希土類元素Rはイットリウム（Y）を包含し、軽希土類及び重希土類を包含する希土類元素である。Rとしては、軽希土類をもって足り、特にNd，Prが好ましい。また通常Rのうち1種もって足りるが、実用上は2種類以上の混合物（ミッシュメタル、ジジム等）を入手上の便宜等の理由により用いることができ、Sm，Y，La，Ce，Gd等は他のR、特にNd，Pr等との混合物として用いることができる。なお、このRは純希土類元素でなくてもよく、工業上入手可能な範囲で製造上不可避な不純物を含有するものでも差し支えない。

【0027】Rは、R－Fe－B系永久磁石を製造する合金鋳片の必須元素であって、10原子％未満では高磁気特性、特に高保磁力が得られず、25原子％を越えると残留磁束密度（Br）が低下して、すぐれた特性の永久磁石が得られない。よって、Rは10原子％～25原子％の範囲とする。

【0028】Bは、R－Fe－B系永久磁石を製造する合金鋳片の必須元素であって、2原子％未満では高い保磁力（iHc）は得られず、15％原子を越えると残留磁束密度（Br）が低下するため、すぐれた永久磁石が得られない。よって、Bは2原子％～15原子％の範囲とする。

【0029】Feは、R－Fe－B系永久磁石を製造する合金鋳片の必須元素であって、60原子％未満では残留磁束密度（Br）が低下し、88％原子を超えると高い保磁力が得られないので、Feは60原子％～88原子％に限定する。また、Feの一部をCo、Niの1種又は2種で置換可能であり、これは永久磁石の温度特性を向上させる効果及び耐食性を向上させる効果が得られるためであるが、Co、Niの1種又は2種はFeの50％を越えると高い保磁力が得られず、すぐれた永久磁石が得られない。よって、Co、Niの1種又は2種の置換量はFeの50％を上限とする。

【0030】この発明による合金鋳片において、高い残留磁束密度と高い保磁力を共に有するすぐれた永久磁石を得るためには、R12原子％～16原子％、B4原子

％～１２原子％、Ｆｅ７２原子％～８４原子％が好ましい。また、この発明による合金鋳片は、Ｒ、Ｂ、Ｆｅの他、酸素、Ｃ、Ｃａ、Ｍｇなどの工業的生産上不可避的不純物の存在を許容できるが、Ｂの一部を４．０原子％以下のＣ、３．５原子％以下のＰ、２．５原子％以下のＳ、３．５原子％以下のＣｕのうち少なくとも１種、合計量で４．０原子％以下で置換することにより、磁石合金の製造性改善、低価格化が可能である。特に、前記Ｂの一部を４．０原子％以下のＣで置換することにより、焼結磁石の耐食性が向上する。

【００３１】さらに、前記Ｒ、Ｂ、Ｆｅ合金あるいはＣｏを含有するＲ－Ｆｅ－Ｂ合金に、９．５原子％以下のＡｌ、４．５原子％以下のＴｉ、９．５原子％以下のＶ、８．５原子％以下のＣｒ、８．０原子％以下のＭｎ、５原子％以下のＢｉ、１２．５原子％以下のＮｂ、１０．５原子％以下のＴａ、９．５原子％以下のＭｏ、９．５原子％以下のＷ、２．５原子％以下のＳｂ、７原子％以下のＧｅ、３５原子％以下のＳｎ、５．５原子％以下のＺｒ、５．５原子％以下のＨｆのうち少なくとも１種添加含有させることにより、永久磁石合金の高保磁力が可能になる。この発明のＲ－Ｆｅ－Ｂ系永久磁石において、結晶相は主相が正方晶であることが不可欠であり、特に、微細で均一な合金粉末を得て、すぐれた磁気特性を有する焼結永久磁石を作製するのに効果的である。

【００３２】この発明において、樹枝状あるいは柱状結晶とＲ－リッチ相とが微細に分散した均質組織を有する磁石合金鋳片の板厚を０．０１mm～１０mmに限定した理由は、０．０１mm未満では急冷効果が大となり、結晶粒径が３μmより小となり、粉末化した際に酸化しやすくなるため、磁気特性の劣化を招来するとともに、微粉砕後の粒子が多結晶となり配向度が低下しＢｒが低下するので好ましくなく、また１０mmを越えると、冷却速度が遅くなり、α－Ｆｅが晶出しやすく、結晶粒径が大となり、Ｎｄリッチ相の遍在も生じるため、磁気特性、特に保磁力が低下するので好ましくないことによる。より好ましくは板厚０．０５mm～０．８mmである。

【００３３】この発明のストリップキャスティング法により得られた特定組成のＲ－Ｆｅ－Ｂ系合金の断面組織は、主相の$R_2Fe_{14}B$結晶が従来の鋳型に鋳造して得られた鋳塊のものに比べて、約１／１０以上も微細であるが、前述のごとく短軸結晶粒径が１．０μm未満の微細結晶を１０％以下含有する平均短軸結晶粒径が３μm～１５μmである。

【００３４】

【実施例】

実施例１

Ａｒ減圧２００ｔｏｒｒ雰囲気で溶湯温度１３００℃の３１Ｎｄ－１．０Ｄｙ－１．１Ｂ－３．０Ｃｏ－残Ｆｅ（ｗｔ％）組成（液相線温度１１７０℃）の合金溶湯を、ノズルより回転数１２０ｒｐｍの外径３００μmの水冷Ｃｕ片ロール表面に、１次冷却速度5×10^3℃／ｓｅｃにて鋳片温度８００℃に冷却後、ロール離脱後に急冷ロールと鋳片収容箱間（距離８m）で鋳片の上下から圧力５ｋｇ／cm^2、流量５００ｌ／ｍｉｎのＡｒガスを吹きつけ、さらに鋳片収容箱内にて圧力５ｋｇ／cm^2、流量５００ｌ／ｍｉｎのＡｒガスを吹きつけ、鋳片を６００℃（固相線温度６６０℃）まで２００℃／ｍｉｎの２次冷却速度にてガス冷却して厚み０．３８mmの鋳片を得た。

【００３５】得られた鋳片の断面を鏡面研摩して光学顕微鏡（倍率４００倍）で観察し、結晶５００個について短軸結晶粒径を線分法にて測定した結果、短軸結晶粒径が１．０μm以下の微細結晶を３％含有の平均短軸結晶粒径４．５μmの$R_2Fe_{14}B$型樹枝状結晶と５μm以下のＲ－リッチ相が微細に分散した均質組織を有していた。得られた鋳片を粗粉砕後、ジェットミル粉砕にて微粉砕して平均粉末粒径３．０μmの微粉末を得た。前記粉末を磁場強度１５ｋＯｅにてプレス圧１ｔｏｎ／cm^2にて成型後、真空にて１０４０℃に４時間焼結後、６００℃に１時間の時効処理を行い、得られた試験片の磁気特性及び平均結晶粒径を表１に示す。

【００３６】比較例１

実施例１と同一組成の合金溶湯を用い、実施例１と同一ロールを使用し、１次冷却速度７５００℃／ｓｅｃにて冷却し、ロール離脱時の鋳片温度は６３０℃であった。さらに、ロール離脱後の鋳片を２００℃／ｍｉｎの２次冷却速度にてガス冷却して鋳片厚０．３０mmの鋳片を得た。得られた鋳片の実施例１と同一方法にて短軸結晶粒径を測定した結果、短軸結晶粒径１μm以下の微細結晶を１８％含有の平均短軸結晶粒径３．２μmの$R_2Fe_{14}B$型樹枝状結晶を得た。得られた鋳片を平均粉末粒径２．９μmに微粉砕する以外は実施例１と同一条件にて焼結磁石を得た。磁気特性及び平均結晶粒径の測定結果を表１に示す。

【００３７】比較例２

実施例１と同一組成の合金溶湯を用い、実施例１と同一ロールを使用し、１次冷却速度１６００℃／ｓｅｃで冷却し、鋳片温度は１１００℃であった。さらに、ロール離脱後の鋳片を６００℃まで１００℃／ｍｉｎの２次冷却速度でガス冷却して鋳片厚０．４３mmの鋳片を得た。実施例１と同一方法にて短軸結晶粒径を測定した結果、短軸結晶粒径１μm以下の微細結晶は０％であったが、平均短軸結晶粒径は３２μmであった。得られた鋳片を平均粉末粒径３．２μmに微粉砕する以外は実施例１と同一条件にて焼結磁石を得た。磁気特性及び平均結晶粒径の測定結果を表１に示す。

【００３８】比較例３

実施例１と同一組成の合金溶湯を用い、実施例１と同一

のロールを使用し、２次冷却速度を２０℃／ｍｉｎにする以外は実施例１と同一の製造条件にて鋳片厚０．３８μｍの鋳片を得た。実施例１と同一方法にて短軸結晶粒径を測定した結果、短軸結晶粒径１μｍ以下の微細結晶は０．５％であったが、平均短軸結晶粒径２１μｍであった。得られた鋳片を平均粉末粒径３．４μｍに微粉砕する以外は実施例１と同一条件にて焼結磁石を得た。焼結磁石の磁気特性及び平均結晶粒径の測定結果を表１に示す。

【００３９】比較例４

実施例１と同一組成の合金溶湯、及び同一のロールを使用し、２次冷却速度２５０℃／ｍｉｎで７５０℃までガス冷却した後、６００℃まで２０℃／ｍｉｎで冷却する以外は実施例１と同一の製造条件にて鋳片厚０．３９μｍの鋳片を得た。実施例１と同一方法にて短軸結晶粒径を測定した結果、短軸結晶粒径１μｍ以下の微細結晶は０．８％であったが、平均短軸粒径は１８μｍであった。得られた鋳片を平均粉末粒径３．３μｍに微粉砕する以外は実施例１と同一条件にて焼結磁石を得た。得られた焼結磁石の磁気特性及び平均結晶粒径の測定結果を表１に示す。

【００４０】

【表１】

	焼結磁石の磁気特性			焼結磁石の平均結晶粒径
	Br (kG)	(BH)max (MGOe)	iHc (kOe)	
実施例1	13.26	42.6	16.4	6.2μm
比較例1	12.80	39.7	15.8	6.3μm
比較例2	13.26	42.6	13.4	12.8μm
比較例3	13.24	42.5	13.7	10.1μm
比較例4	13.22	42.4	14.2	9.6μm

【００４１】

【発明の効果】この発明は、Ｒ−Ｆｅ−Ｂ系合金溶湯を真空溶解炉にて溶解した後、タンディシュ先端部のノズルより急冷ロールに注湯し、溶湯を急冷ロールにて特定の冷却速度にて１次冷却後、ロールより離脱した鋳片を固相線温度以下に特定の冷却速度にて２次冷却することにより、特定寸法の短軸結晶粒径を有する$R_2Fe_{14}B$型樹枝状結晶あるいは柱状結晶と特定のＲリッチ相とが微細に分散した均質組織からなる特定厚の急冷鋳片を得るもので、配向度の低下及び磁石化の際の粉砕時の微粉化、粉末の酸化を防止でき、磁気特性の優れたＲ−Ｆｅ−Ｂ系磁石合金鋳片が得られる。

フロントページの続き

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